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Film Stress and Adhesion Characteristics of Passivation layers for Thermal ink-jet printhead

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ABSTRACT

Amorphous, hydrogenated silicon carbide (a-SiC:H) deposited by plasma-enhanced-chemical-vapor-deposition (PECVD) has been used as the most important film of passivation layers in a thermal ink-jet printhead. When the printhead was thermal-cycled from room temperature to about 400°C, the a-SiC : H film is sustained by a variety of thermal and mechanical stresses that are detrimental to it's integrity. Thermal stress changes of a-SiC:H films were varied with different CH_4/SiH_4 gas ratios. Microstructure investigation was mainly achieved by FTIR technique. Less Variation of the Si-H absorption bond causes less thermal stress change.

Thin-film adhesion is an important problem in thermal ink-jet printhead between the Ta thin film and a-SiC:H films. A qualitative measure of film adhesion can be made with the scratch tester. The adhesive critical load and Ta coating failure modes on a-SiC:H were acquired to examine the film adhesion of these two investigated films. The adhesion depends on the nature of the interfacial region, which depends on the interactions between the depositing Ta thin film and the surface a-SiC : H films. An increased effective contact area in the interfacial region promotes a good adhesion.

Keyword: Thermal ink-jet printhead, a-SiC:H, thermal stress changes, film adhesion

1. INTRODUCTION

The printhead resistor structure for thermally exciting the ink ejection is fabricated on a silicon (Si) substrate using standard IC processing steps. [1] Silicon oxide is deposited on the Si substrate as a barrier layer to prevent leaching of impurities. The resistor and conductive layers are tantalum (Ta)-aluminum (Al) and Al, respectively. The resistor-conductor films are lithographically patterned to form individual heater and conductive stripe line. Then heater and conductor are covered with ink-resistant passivation films. A dry-film coating further protects the passivation and the underlying thin films from degradation by the ink. To improve contact reliability, the Al pads are coated with Ta and gold (Au) films. A thin film resistor film is rapidly heated through a joule-heating process by direct-current (d.c.) electrical pulses which pass through the resistor and each time last a few microseconds. [2] The temperature the top surface of the device is raised, instantaneously, to over 300℃. The ink-vapor bubble formed adjacent to the resistor propels an ink droplet out of nozzle to form a dot on the paper. After the electrical pulse is turned off, the vapor bubble collapses, subjecting the thin-film passivation to severe hydraulic forces. During the operation and life of the printhead, the passivation experiences sever electrical, thermal, mechanical, and chemical stresses. Developing a passivation film for these exacting requirements presented some interesting challenges. In the present printhead structure, in its operation mode, is thermally cycled between room temperature and 300-plus ℃, at repetition rates of several kHz. In this respect, thermal stress in each layer due to thermal expansion mismatch is calculated. Chang et al. [2] have showed that stresses in the carbide (SiC) and resistor layers due to thermal expansion mismatch may approach 10^{10} dyne/cm^2 under normal condition. Therefore, thermal stress changes of SiC film play an important role for the thermal printhead. Windischmann [3] has reported that the as-deposited SiC films are in compression with absolute values at high as 2×10^{10} dyne/cm^2. The origin of the stress is attributed to hydrogen incorporation, as evidenced by C-H and Si-H bands observed in infrared transmission measurements. Microhardness and scratch adhension testing are the most commonly used techniques for assessing the mechanical properties of thin surface coatings. Burnett et al [4] have reported schematic representation of coating failure modes in the scratch test. They revealed different film adhension force results in different failure modes.

In the present study, thermal stress changes for SiC film deposited by PECVD in different gas ratios were investigated. They are correlated with variations of chemical bonding structure for SiC films subtracted out by infrared spectrum (FTIR). Adhension characteristics of passivation layers, tantalum (Ta) and SiC, played an important role in the printhead thin film

structure. Scratch testing was performed to find out Ta film coating adhension on the different depositing conditions of SiC films.

2. EXPERIMENTAL PROCEDURE

Amorphous SiC films were prepared on silicon wafers of 4-inches diameter. The film were deposited in a capacitively coupled, parallel plate rf glow discharge apparatus. The system (SLR-730, Plasma Therm Inc., FL) has a four-wafers load-lock system, including 125 kHz low frequency (LF) and 13.56 MHz high frequency (HF) rf generators. Deposition experiments were performed at a temperature of 350℃, a power of 100 w, a pressure of 1300 mT and using CH4/SiH4 gas mixtures with helium (He) as a gas carrier. Table I indicates the gas flow ratio of CH4/SiH4, and other different processing parameters for SiC films using SLR-730 system. Film thickness was measured by the prism coupler. (Metricon Model 2010) Film stress was calculated from wafer curvature measured on the laser-based type at the dual wavelength of 670 nm and 750 nm (FLX-2320, Tencor). Thermal stress changes of SiC films were performed in the nitrogen atmosphere from room temperature to 450℃ at a rate of 7.5℃ /min, then cooled in the same gas for six thermal cycles. Si-H concentrations were computed from infrared spectra (FTIR, with the Si substrate premeasured and subtracted out).
The adhension strength of Ta coating (600 nm) on SiC films prepared by different gas ratios from Table 1 at a high frequency (13.56 MHz) were measured by the scratch tester. For distinguishing, scratch testing on Ta over SiC film deposited by a low frequency (125 KHz) was conducted. Parameters for the scratch tester are including of 125 um diamond head, scratch length at 0.3~1.0 cm, loading speed at 1.7 Nt/sec, and the maximum loading force at 10~20 Nt.

3. RESULTS AND DISCUSSION

3.1 DEPOSITING CHARACTERISTICS OF A-SIC:H FILM

Fig.1 shows that deposition rate of a-SiC:H films decreases by increasing CH4/SiH4, gas ratio. In this experiment, the system is performed at a low operating power region, therefore the ionized threshold for the CH4 gas is not reached. CH4 gas molecules would absorb supplied power from PECVD instead of forming reactive radicals. Therefore higher gas ratio caused lower deposition rate for a-SiC:H films.

3.2 CHEMICAL BONDING STRUCTURE OF A-SIC:H FILM

Fig.2 shows the typical absorption spectrum for a-SiC:H film measured by FTIR. It indicates those four possible chemical bonds for every absorptive peak on a typical infrared spectrum. For the SiH_n absorptive peak, wave number showed a little shift ranging from 2000 to 2140 cm^{-1} when carbon compositions wt% increased, as shown in Fig.3. This result is caused from different electron negativity between Si and C.[5] In Si-rich region, SiH_n reveals Si-H or $Si-H_2$ bonding; but in C-rich region, SiH_n reveals preferentially Si-H bonding. $Si-H_n$ bonding concentration can be calculated from the following equation: [6,7]

$$N = A_s \int \frac{\alpha(\nu)}{\nu} d\nu \approx \frac{A_s}{\nu_0} \int \alpha(\nu) d\nu$$

Where As is the inverse absorption cross section of the considered mode, ν_0 is the wave number corresponding to the absorption peak, $\alpha(\nu)$ is absorption coefficient. Fig.4 shows Si-H concentration variations with different gas ratios for as deposited and after six thermal cycles.

3.3 STRESS ANALYSIS OF A-SIC:H FILM

The total stress in the structure is made up of two terms, i. e., stress due to differential thermal expansion effects and that due to intrinsic stress, such as stress due to argon incorporation, impurity. [2] Thermal stress is resulted from the thermal expansion mismatch effects between a-SiC:H film and Si substrate. Intrinsic stress is due to chemical bonding structure within different compositions of a-SiC:H films. Variations of bonding structure are dependent on different gas ratios. Fig.5 (a) and (b) reveal thermal stress changes of two different gas ratios of a-SiC:H films are performed in the N_2 atmosphere from the room temperature to 450℃. According to the first thermal cycle, stress change is inclined to more compressive stress until 350℃, this result is due to bigger thermal expansion coefficient of a-SiC:H film than Si substrate. Above 350℃, the stress changes toward more tensile according to shrinking of SiC films forming a condense film structure. Therefore the intrinsic stress causes substantially total stress changes toward tensile stress when the temperature is back to the room

temperature. Fig.5 also indicates that total stresses are inclined to saturation according the small shrinking of SiC films after six thermal cycles. Fig.6 shows that total stresses change toward tensile stresses for every a-SiC:H films deposited from different gas ratios on the Table I. Fig.7 indicates that stress changes after six thermal cycles decrease as CH4/SiH4 gas ratios increased. Thermal stress changes are correlated with chemical bonding structure investigated by FTIR technique. Variations of the Si-H concentration for a-SiC:H films with different gas ratios are shown in Fig.8. Less variation of the Si-H absorption bond causes less thermal stress change.

3.4 TA/SIC FILM ADHENSION

Thin-film adhesion is an important problem in thermal ink-jet printhead between the Ta thin film and a-SiC:H films. A qualitative measure of film adhesion can be made with the scratch tester. The adhesive critical load and Ta coating failure modes on a-SiC:H were acquired to examine the film adhesion of these two investigated films. Fig.9 (a) reveals the optical microscopic image for Ta coating over a-SiC:H prepared by a gas ratio of 90 and high frequency of 13.56 MHz . Nevertheless, acoustics emission signal acquired from the scratch tester with scratch length on Ta film is shown in Fig.9 (b). It reveals that critical load is 3.5 Nt. In the present study, critical loads for Ta coating over a-SiC:H films deposited at a high frequency shown in Table I are ranged from 1.6 to 3.5Nt. Failure mode on Ta coating over a-SiC:H film by scratch tester, as shown in Fig.9(a), reveal a crack with regular chipping failure mode, it is a kind of delaminating phenomena. [4] In order to enhance film adhesion of these two investigated films, low frequency power (125 kHz), working pressure (900 mT), and the same gas ratio are used to deposit a-SiC:H film, then the same Ta film coating on it. Fig.10 (a) reveals the optical microscopic image for Ta coating over a-SiC:H prepared by low frequency power, failure mode on Ta is tensile cracking when the coating remains fully adherent. Fig.10 (b) shows acoustics emission signal with the scratch length on Ta film and critical load reaches to 18 Nt as compared with optical microscopic images, shown in Fig.10 (a). Thin-film adhension was affected by two factors: one is bonding force between Ta and a-SiC:H films, the other is the effective contact area of the interface. Hey et. al. [8] reported that ion bombardment of the a-SiC:H film prepared by low frequency and working pressure can result in more condense film. Fig.11 (a) and (b) show infrared spectra of the high frequency and low frequency a-SiC:H films at the same gas ratio, respectively. Obviously, Si-H and C-H bonds are rarely found out in infrared spectrum of a-SiC:H film by a low frequency. The adhesion depends on the nature of the interfacial region, which depends on the interactions between the depositing Ta thin film and the surface a-SiC：H films. An increased effective contact area in the interfacial region promotes a good adhesion.

CONCLUSIONS

Higher gas ratio, CH_4/SiH_4, caused lower deposition rate for a-SiC:H films. Thermal stress changes of a-SiC:H films were examined in the nitrogen atmosphere from room temperature to 450℃ at a rate of 7.5℃ /min, then cooled in the same gas for six thermal cycles. Thermal stress changes are correlated with chemical bonding structure investigated by FTIR technique. Less variation of the Si-H absorption bond causes less thermal stress change. a-SiC：H films are sustained by a variety of thermal and mechanical stresses that are detrimental to it's integrity. Developing a passivation film for these exacting requirements presented some interesting challenges. Thin-film adhesion is an important problem in thermal ink-jet printhead between the Ta and a-SiC:H passivation films. An increased effective contact area in the interfacial region promotes a good adhesion.

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Table I Processing parameters of a-SiC:H film

CH_4/SiH_4	3	6	12	15	30	45	60	90
SiH_4 flow rate (sccm)	450	450	450	450	450	300	225	150
CH_4 flow rate (sccm)	67.5	135	270	337.5	675	675	675	675
He flow rate (sccm)	782.5	715	580	512.5	175	325	400	475
Depositing time (min)	100	100	100	100	100	100	100	150
Film thickness (nm)	1794.6	1014.5	1015	940	1051.5	503.2	633.7	853.3

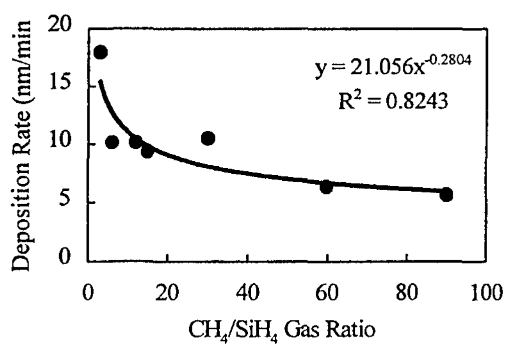


Fig. 1 Deposition rate of a-SiC:H films deposited by different CH_4/SiH_4, gas ratio

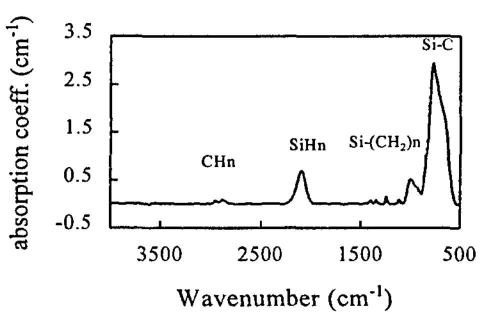


Fig.2 Typical absorption spectrum for a-SiC:H film measured by FTIR

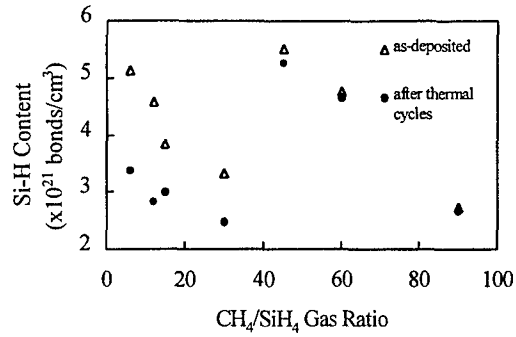


Fig. 3 Si-H_n wavenumber vs carbon% in a-SiC:H films

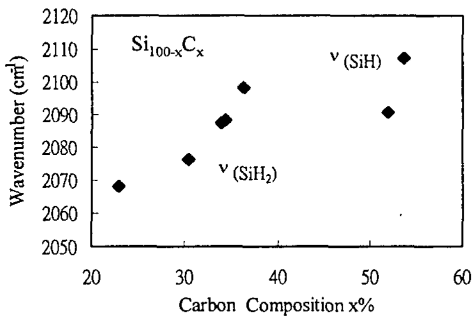


Fig. 4 Si-H concentration vs CH_4/SiH_4 gas ratio

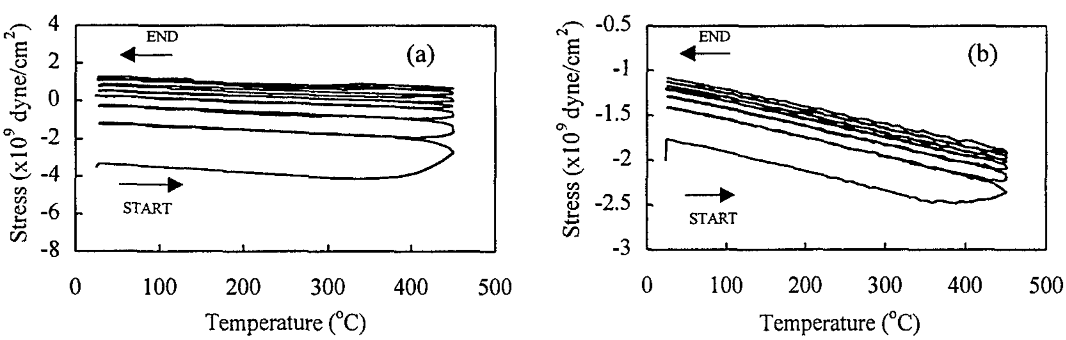


Fig.5 (a) Thermal stress curve for a-SiC:H film (gas ration=6) and (b) Thermal stress curve for a-SiC:H film (gas ration=90)

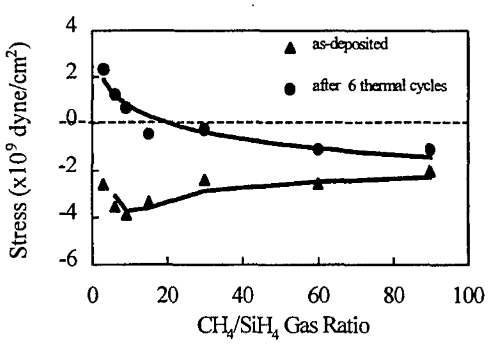


Fig. 6 Film stress before and after 6 thermal cycles for a-SiC:H films prepared by different gas ratios

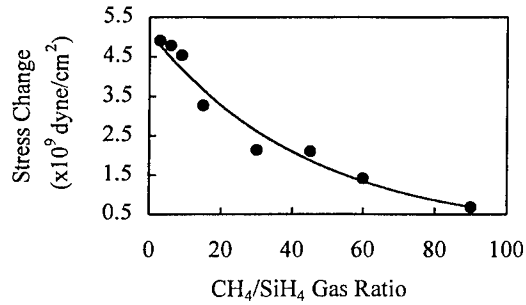


Fig.7 Stress changes after 6 thermal cycles for different gas ratios

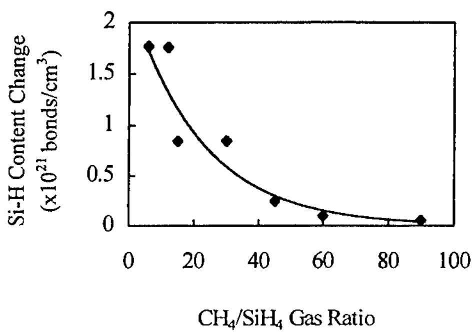


Fig. 8 Si-H concentration change after 6 thermal cycles for different gas ratio a-SiC:H films

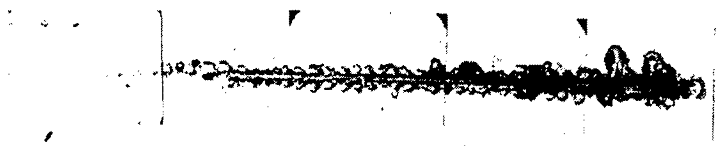


Fig.9 (a) Optical microscopic image for Ta scratch testing on high frequency a-SiC:H film

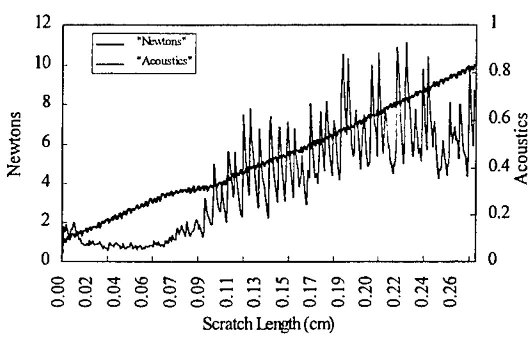


Fig.9 (b) Acoustics emission signal vs scratch length for Fig. 9(a)

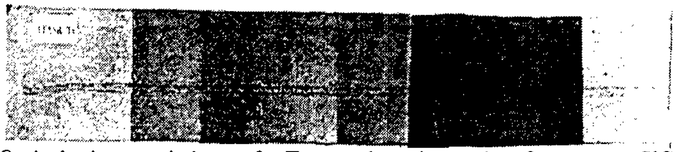


Fig. 10 (a) Optical microscopic image for Ta scratch testing on low frequency a-SiC:H film

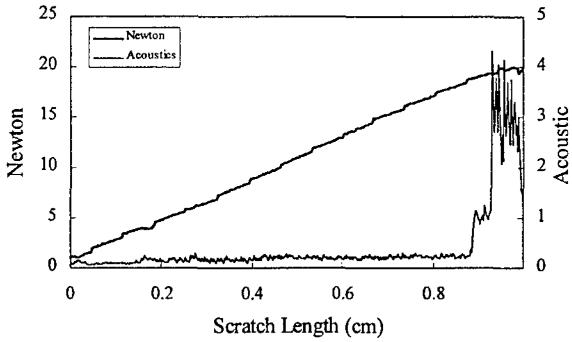
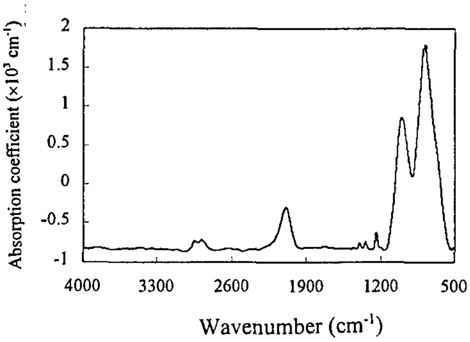


Fig .10 (b) Acoustics emission signal vs scratch length for Fig. 10 (a)



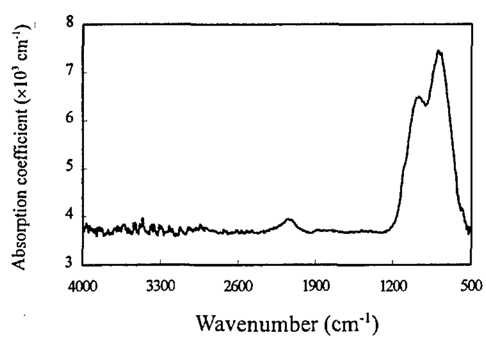


Fig .11 (a) FTIR spectrum for a-SiC:H film deposited by high frequency, gas ratio=12
(b) FTIR spectrum for a-SiC:H film deposited by low frequency, gas ratio=12